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I, JANENE PEISKER, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2004907247 for a patent by WINE NETWORK TECHNOLOGY PTY. LTD. as filed on 21 December 2004.



WITNESS my hand this
Twentieth day of June 2005

A handwritten signature in black ink, appearing to read 'J.K. + C.'.

JANENE PEISKER
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2004907247 21 Dec 2004

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WINE NETWORK TECHNOLOGY PTY. LTD.

A U S T R A L I A

Patents Act 1990

PROVISIONAL SPECIFICATION

for the invention entitled:

Alcohol Reduction in Beverages

The invention is described in the following statement:

ALCOHOL REDUCTION IN BEVERAGES

The level of alcohol in beverages such as wine is an important determinant of its perceived quality. It is, in turn, largely determined by the level of sugar in the grapes from which it is produced. Low levels of alcohol are commonly associated with grapes grown in cooler climates or seasons. Less positively they are also a result of under-ripe or over-irrigated grapes and in these instances are seen as an indicator of low quality wine. High levels of alcohol are, as a result, deemed to be associated with fully ripe fruit and higher quality. This is not a consequence of the higher alcohol per se but rather the more mature fruit flavours, tannins and lower acidity of grapes picked at optimum ripeness. In fact the pursuit of greater ripeness by winemakers in many parts of the world has resulted in wines with excessive alcohol. Besides increasing the intoxicating effect of the wine, this manifests itself in a reduced perception of wine aroma as well as an unpleasant hotness on the palate.

A measure of the extent of this problem shows it is growing at a worrisome rate. Wine samples analysed by the Australian Wine Research Institute over the past 20 years have shown a steady increase in alcohol level over this period so that the mean for all samples analysed in 2002 was 14.2% compared with 12.4% in 1984. These elevated alcohol levels can have other damaging effects on wine quality such as prolonged or arrested primary and secondary fermentations, leading to higher levels of residual sugar, with consequent microbiological spoilage, loss of SO₂ and oxidation. (AWRI 2003 Annual Report p44).

A method for removing some of this alcohol would allow winemakers to pick their grapes at optimum ripeness from the point of view of flavour maturity without suffering the negative effects of excessive alcohol.

Processes for reducing alcohol have been offered previously but all are deficient in some way.

The simplest method for reducing alcohol is to add water to the grape must or wine. While this has been practised for centuries, it diminishes wine quality by reducing the overall concentration of the wine. It is also illegal in many jurisdictions.

5 A more effective procedure is to remove alcohol using a low temperature distillation technique such as the spinning cone. In this, volatile components of the wine, including alcohol, are removed in the distillate and the volatile flavours are separated from this and returned to the wine being treated. This system is complex, capital intensive and immobile. There is also some possibility of flavour loss, but most importantly, the alcohol
10 is removed at relatively low strength (<50% v/v) so overall volume loss from the wine is significant.

Another technique is proposed in Patent Specification No. AU B 42319/93. In this proposal wine is processed through a reverse osmosis plant to generate a permeate stream
15 which consists substantially of water, alcohol and low concentrations of some other minor components. The permeate stream is then distilled in a high energy distillation column and the distillate which consists very substantially of high strength alcohol, is removed as a useful by-product. The residual material, being dealcoholised permeate, is returned to the wine, thus reducing its alcohol content. This is effective but costly in energy terms as well
20 as infrastructure costs.

According to Williams *Williams L. Distilled Beverage Technology, course notes, UC Davis 1981*, "Because of this non-ideality, the relative volatility of ethanol with respect to water varies greatly. It is very large (10 to 11) in dilute solutions and decreases to 1.0 at
25 the azeotropic concentration. ... Thus alcohol enrichment is very large at low concentrations and one may say that distillation is "easily achieved" in this region. At high alcohol concentrations, the enrichment is small and thus, distillation to very high concentration is "difficult or costly" (in terms of energy, equipment size or both)."

30 As well, in many jurisdictions distillation is strictly regulated because of the inherently hazardous nature of the high strength alcoholic spirit produced as well as its

2004907247 21 Dec 2004

2004907247 21 Dec 2004

interest to taxation authorities for excise revenue purposes. This means that the distillation process must be conducted in licensed premises which are usually remote from the wine being processed. This necessitates the wine or permeate being shipped from the winery to the distillation premises and the dealcoholised permeate being returned. Besides the
5 freight costs and delays of this, in some jurisdictions it is mandatory for the dealcoholised permeate to be recombined only with the wine from which it was originally removed. This means batches must be handled discretely, reducing the prospects of scale economies and, more importantly, the dealcoholised permeate is microbiologically unstable and will quickly deteriorate unless preserved by refrigeration or chemical stabilisers.

10

Another option practised in jurisdictions where this is allowed, is to remove a certain amount of permeate by reverse osmosis and to replace it with the same amount of water. This water could be from grape or non-grape sources according to the local regulations but in most wine producing countries the practice is illegal or of dubious status.

15 Another deficiency of this approach is that the permeate which is discarded does contain some other, minor components that would be lost and so the quality of the wine may be slightly diminished.

An approach described by *Hogan et al: Osmotic Distillation Chemical Engineering Progress 1997* and *A New Option: Osmotic Distillation, Chemical Engineer Progress July 1998* uses the process of evaporative perstraction to remove alcohol from wine. This technique is also disclosed in Patent Specification No. AU 199717793 B2 and involves passing a stream of wine through a membrane contactor such as a Liqui-Cel® Extra-Flow produced by Membrana. Separated from the wine stream by an hydrophobic membrane, a
25 counterflow of water is passed through the same contactor and alcohol passes through the membrane from the wine to the water. This process is based on the principle that ethanol, as a volatile wine component, has a significant vapour pressure. This leads to its movement into the porous matrix of the hydrophobic membrane and by virtue of the concentration difference across the membrane, its subsequent dissolution into the water on
30 the other side.

In practice this results in high levels of extraction of other desirable volatile components from the wine, such as flavours, esters and sulphur dioxide. One approach suggested by the developers of this technique was to "spike" the strip solution with these compounds so that no concentration gradient for the compound exists. This is complex and expensive and renders the by-product less useful. Other efforts to limit the extraction of desirable volatiles by recycling some of the strip stream reduce the efficiency of the process. Efficiency is also compromised by the presence of relatively large concentrations of CO₂ and other gases in wine. These cannot easily be removed without also removing desirable volatiles.

Australian Patent Application No. 2004903139 discloses a technique of alcohol reduction in an alcohol containing beverage which includes the steps of treating the beverage by reverse osmosis or nanofiltration and subjecting the permeate to a stripping solution in order to remove alcohol therefrom and then returning the dealcoholised permeate to the retentate to thereby reduce the overall alcoholic content of the beverage. The content of the aforementioned specification is incorporated herein by reference. Whilst the technique disclosed in that application produces satisfactory results, it has been found in accordance with the invention that the techniques can be used by carrying out the stripping at an elevated temperature.

Normally volatile components in wine are destroyed if the wine is heated. In the process of the invention, however, the wine itself is not subjected to elevated temperatures but rather the permeate only is subjected to elevated temperatures. Accordingly, superior alcohol stripping can be achieved without degradation of the components in the wine which give it taste and aroma. Stripping at elevated temperatures is much more efficient than stripping at lower temperatures. In the aforementioned article by *Hogan et al.*, the stripping is necessarily carried out at low temperature otherwise the properties of the wine would be seriously downgraded. Accordingly, the process of the invention is more efficient than that described in the aforementioned article.

The object of the present invention is to provide an improved technique of dealcoholisation of beverages which minimises extraction of desirable volatile components from the beverage.

5 According to the present invention there is provided a method of reducing the alcohol content of an alcohol containing beverage including the steps of:

- (i) processing the beverage by reverse osmosis or nanofiltration for producing a retentate and a raw permeate which includes alcohol;
- (ii) contacting a first side of an hydrophobic microporous membrane with said
10 heated raw permeate;
- (iii) contacting a second side of the membrane with a heated strip solution to extract alcohol therefrom to form a dealcoholised permeate; and
- (iv) combining the retentate with the dealcoholised permeate to thereby reduce the alcoholic content of said beverage.

15

Preferably, the strip solution and/or the raw permeate are heated prior to contacting the microporous membrane. It is further preferred that the strip solution and raw permeate are both heated prior to contacting the porous membrane. It will be appreciated that there will be heat conduction between the permeate and strip solution if they are not at the same
20 temperature and therefore it would be possible, although less desirable, to heat one or other of these solutions.

Preferably, the strip solution and/or the raw permeate has a temperature in the range from 40°C to 70°C and most preferably about 45°C to 50°C when in contact with
25 said microporous membrane.

Normally the dealcoholised permeate will be at approximately the same temperature as the strip solution and preferably the method includes the step of cooling the dealcoholised permeate prior to recombining it with the retentate.

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In the process of the invention, the beverage itself is not subjected to evaporative perstraction but rather the alcohol rich permeate is subjected to the evaporative perstraction. The beverage is also not subjected to elevated temperatures.

5 Preferably the step of processing the beverage by reverse osmosis or nanofiltration is maintained at a temperature in the range from 13°C to 25°C.

Where the beverage is wine, the extraction of volatiles is reduced because of their limited passage from the wine into the permeate stream. This is controlled by the selection
10 of appropriate membranes and operating parameters such as temperature, pressure and flow rate to maximise the passage of ethanol while limiting the passage of other compounds.

Further, the efficiency of the evaporative perstraction process is improved by
15 reducing the concentration of non-condensable gases in the membrane headspace. Trials and modelling of the process based on known vapour pressures of the gases, ethanol and water, suggest significant efficiency gains in terms of ethanol removal for given surface areas of membranes.

20 Efficiency of perstraction can be improved by reducing gas concentrations in the product and strip feeds.

The strip solution preferably is purified water. The water may be purified by reverse osmosis or particulate and carbon filtration.

25

Preferably further, the raw permeate is processed so as to remove oxygen and carbon dioxide and nitrogen therefrom prior to contacting the permeate with the microporous membrane.

30 Preferably further, the water also has oxygen, nitrogen and carbon dioxide removed therefrom prior to contacting with the membrane.

2004907247 21 Dec 2004

The alcohol in the strip solution is a useful by-product.

The invention also provides apparatus for reducing the alcohol content of an alcohol containing beverage, the apparatus including:

- 5 (i) a first processing stage having a reverse osmosis unit or nanofiltration unit having a retentate outlet and permeate outlet;
- (ii) a pump for supplying beverage to be treated under pressure to the first processing stage whereby retentate is produced at the retentate outlet and raw permeate containing alcohol is produced at the permeate outlet;
- 10 (iii) a second processing stage which includes at least one hydrophobic microporous membrane, means for contacting heated raw permeate to one side of said membrane and means for contacting a heated strip solution to the other side of the membrane whereby the membrane is operable to remove at least a portion of the alcohol from the raw permeate so as to produce dealcoholised permeate at the outlet of the second
- 15 processing stage; and
- (iv) means for combining said dealcoholised permeate with said retentate to thereby produce treated beverage in which the alcoholic content thereof is reduced.

20 Preferably, the apparatus includes heater means for heating the strip solution and/or the raw permeate to a temperature in the range from 40°C to 70°C and most preferably to a temperature of about 45°C to 50°C.

25 Preferably further, the apparatus includes means for cooling the dealcoholised permeate prior to combining with said retentate.

The invention will now be described with reference to the accompanying drawing which shows a schematic is a schematic block diagram of a system for reducing the alcoholic content of wine.

30 The diagram schematically illustrates a system 2 for producing reduced alcohol wine in accordance with the invention. The system 2 includes a tank 4 for storage of wine

- 8 -

to be treated. Wine from the tank 4 passes to a pump 6 which pumps the wine at high pressure to a reverse osmosis unit 8. The reverse osmosis unit 8 has membranes therein which pass water and alcohol into the permeate while rejecting other desirable wine aroma, colour and taste components which are retained in the concentrated wine or retentate. The reverse osmosis unit 8 has a permeate outlet line 10 and a retentate outlet 12. The outlet 12 is connected by means of a line 14 to the tank 4 for circulating the reduced alcohol wine. The line 14 includes a back pressure control valve 16 which effectively controls the pressure in the reverse osmosis unit 8 and outlet 12. The membranes in the reverse osmosis unit 8 can typically be in the form of spiral wound reverse osmosis or nanofiltration membranes such as GE Osmonics VinoPro 8040 or 4040.

Typically the flow of wine through pump 6 is 3,500 to 12,500 litres per hour, depending on type and number of membranes used.

Typically the temperature and pressure in the reverse osmosis unit 8 and outlet 12 are in the range 13°C to 25°C and 1,500 kPa to 4,000 kPa but the pressure in the line 12 is about 100 to 200 kPa after the back pressure control valve 16.

Normally the percentage of wine passing to the retentate outlet 12 will be in the range 75% to 90%.

Normally the wine in the tank 4 will have an initial alcoholic content in the range from say 13% to 16% by volume. The system of the invention seeks to reduce the alcoholic content of the wine in tank 4 to a more desirable level such as say 12.5% to 13.5%.

Typically the alcoholic level of the permeate at the raw permeate outlet 10 is 10% to 13% v/v. The flow of permeate leaving the reverse osmosis unit 8 is measured in the line 14 by mag flowmeter 194. Its temperature is measured by temperature probe 196. Both of these measurements are transmitted to a separate programmable logic controller (not shown) for display and control.

2004907247 21 Dec 2004

The system of the invention includes a first heat exchanger 18 which warms the permeate by counterflow heat exchange with the hot treated permeate returning in line 62. A line 36 is connected to the first heat exchanger 18 so as to pass the raw, pre-warmed permeate to a second heat exchanger 22 which heats the permeate further.

5

The system includes first, second, third, fourth, fifth and sixth contactors 24, 26, 28, 30, 32 and 34, arranged in a vertical orientation. The contactor 24 is the water degassing contactor. The contactor 26 is the permeate degassing contactor. The contactors 28, 30, 32 and 34 are the alcohol stripping contactors. Each of these can be of the type which includes a hydrophobic microporous membrane, for example of the type Liqui-Cel® Extra-Flow. A line 136 is connected to the second heat exchanger 22 so as to pass the raw, hot permeate to the second contactor 26.

10

The system of the invention also includes a vacuum pump 40, the inlet of which is connected to a vacuum line 42 and includes a non return valve 188 to prevent service water running back into the vacuum line 42. The vacuum line 42 is connected to the second contactor 26 and then to the first contactor 24 by means of a line 46. The first contactor 24 has an inlet line 44 for supplying a counterflow of an inert gas such as nitrogen. Typically the flow of nitrogen is regulated to be about 400 litres per hour.

15

Normally the raw permeate is supplied to the shell side of the contactor 26 whereas the vacuum is applied to the lumen side or the interior of the multiplicity of membrane tubes which pass through the contactors 24 and 26. The vacuum has the effect of removing carbon dioxide and oxygen from the stream of warm stripping water in the contactor 24 and from the stream of warm raw permeate in the contactor 26.

20

The second contactor 26 is connected to the bottom of the third contactor 28 by means of a line 48 so as to input the degassed permeate to the shell side of the contactor 28. The top of contactor 28 receives a degassed strip water solution on input line 150 from the first contactor 24. The alcohol stripping action takes place in the third, fourth, fifth and

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2004907247 21 Dec 2004

sixth contactors 28, 30, 32 and 34 where the degassed permeate encounters a counterflow of the degassed strip water and its alcohol level is typically reduced to 3% to 6% v/v.

In the illustrated arrangement, the stripping contactors 28, 30, 32 and 34 are arranged in two parallel banks so that the stream of degassed permeate entering from the line 48 is split to line 50 before flowing upwards through the contactor 28 then through another line 52 to the bottom of the contactor 30. An isolation valve 51 allows the contactors 28 and 30 to be isolated from the system. Similarly, the permeate in the line 50 flows through an isolation valve 53 and the contactor 32 then through a line 54 to the bottom of the contactor 34. The alcohol reduced permeate passes from the contactor 30 via a line 56 to a flow control valve 58 then to a flow monitoring rotameter 60 to a further line 62. A similar line 64, flow control valve 66 and flow monitoring rotameter 68 pass the alcohol reduced permeate from the contactor 34 to the line 62. A pressure transmitter 70 monitors the back pressure in the permeate lines 48 and 50 and transmits its measurement to a separate controller (not shown) for display.

The relative flows of permeate through the two contactor banks 28, 30 and 32, 34 and the pressure as measured by pressure transmitter 70 are controlled by flow control needle valves 58 and 66.

20

The still warm reduced alcohol permeate then passes through the line 62 to the heat exchanger 18 where it is cooled by, and in turn pre-warms the raw permeate coming from the reverse osmosis unit 8 via permeate outlet line 10. The treated and cooled permeate from the first heat exchanger 18 then passes through a line 72 and non return valve 74 to be mixed with the concentrated wine (retentate) in the line 14 for return to the tank 4. The wine returning to the tank 4 therefore has a reduced alcoholic content which is typically from say 0.5% to 1.5% less than the untreated wine in the tank 4. The flow rate of reduced alcohol permeate is measured by a magnetic flowmeter 190 and its temperature is measured by a temperature probe 192. Where the alcohol reduction in the range from 0.5% to 1.5% is sufficient, the treatment can be carried out in a single pass in which case the line 72 would pass to a separate collection tank for treated wine (not shown).

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21 Dec 2004

2004907247

Normally, however, a greater reduction in alcohol would be required and a number of passes would be carried out in order to give the required overall reduction of alcoholic content of the wine.

- 5 By comparing the temperature corrected flows in the lines 10 and 72, the difference in flows should correlate with the rate of alcohol removed and so provide a means of monitoring and controlling the performance of the alcohol reduction process.

10 In accordance with the invention, the alcohol stripping is carried out on the permeate rather than the wine itself and therefore desirable volatile components in the wine remain substantially unchanged because they remain in the retentate. Importantly, the permeate is raised to elevated temperatures for stripping and the wine is not subjected to elevated temperatures.

- 15 The system includes a water tank 76 which stores water to be supplied via an inlet line 78 to a pressure pump 80. Preferably the water has been purified say by reverse osmosis prior to admission to the tank 76. However, where water quality permits, this could be a mains supply. The pump 80 supplies water from the tank 76 under pressure via a line 82 to a break tank 84 which includes a float valve 86 to maintain a constant level of
20 service or seal water for a vacuum pump 40. The break tank 84 includes a line 88 and valve 90 to drain the tank to a refuse point 92.

- The pressure pump 80 also supplies water via a line 94 to a surge tank 98. Maximum flow to this tank is regulated by a valve 96 and level in the tank is maintained
25 by a float valve 100. An overflow line 102 passes any excess to a drain point 104. A drain line 186 with a valve 184, allows the surge tank 98 to be drained to the point 92 (or 104, if more convenient).

- Water in the surge tank 98 then passes via a line 106 to a pump 108, then on to a
30 combined particulate and adsorbent carbon filter 112. Differential pressure across the filter 112 is monitored by pressure gauges 116 and 118. The purpose of this filter is to remove

- 12 -

any large solids in the water which could foul the membranes in the contactors and any taints which could pass the membranes which might otherwise taint the permeate and then the wine.

5 After the filter 112, the water passes through a line 122 to a third heat exchanger 126. Flow in the line 122 is monitored by a flow detector switch 124 which sends a signal to a separate controller (not shown) in the event of no flow in the line. The water from the line 122 is pre-warmed in the third heat exchanger 126 by a counterflow of hot, alcoholic strip water returning from the stripping contactors via a line 128. The cooled alcoholic
10 strip water leaves the third heat exchanger 126 for recovery or disposal to waste via a line 142. The flow of alcoholic strip water by-product in the line 142 is measured by a flow totaliser 146.

15 The pre-warmed raw strip water from the third heat exchanger 126 then passes through a line 130 to a heater 132 where it is heated to approximately 65°C to 75°C. The heater 132 could be of various types such as a gas heater, electric heater or heat pump which is most appropriate for the site and the duty cycle.

20 The heated strip water then passes through a line 134 to the second heat exchanger 22 where it warms the raw permeate entering in counterflow from the line 36. The heated strip water then passes through a line 38 to the first contactor 24 for degassing. The temperature of the heated strip water in the line 38 is monitored by temperature transmitter 140 which sends an analogue signal to a separate controller (not shown). The heated permeate from the second heat exchanger 22 then passes through a line 136 to the second
25 contactor 26 for degassing. The temperature of the heated permeate in line 136 is also monitored by a temperature transmitter 138. The second heat exchanger 22 is sized so that the counterflows of permeate and strip water both leave heat exchanger 22 at a temperature in the range 40°C to 70°C and most preferably at about 45°C to 50°C. The upper temperature is largely determined by the temperature tolerance of the contactors. It will be
30 appreciated that the heating of the water by the heater 132 and the properties of the heat exchanger 22 together with various flow rates of permeate and retentate in this system,

2004907247 21 Dec 2004

essentially control all of the temperatures throughout the entire system. Normally, the wine in the tank 4 is relatively cool, say between 10°C and 25°C and similarly the water in the tank 76 is also normally in these temperature ranges. The first heat exchanger 18 acts to recover heat and cool the permeate before it is returned to the wine in the tank 4. The
5 third heat exchanger 126 acts to recover heat by preheating the water supplied to the heater 132.

After heating in the second heat exchanger 22 and passing through the line 38, the heated, raw strip water enters the first degassing contactor 24 where the level of dissolved
10 oxygen and carbon dioxide is reduced. The degassed strip water then passes from the contactor 24 via an input manifold line 150 to the tops of the third, fourth, fifth and sixth stripping contactors 28, 30, 32 and 34. A flow control needle valve 110 adjusts the overall flow of strip water and the pressure in the line 150. Isolation valves 152, 154, 156 and 158
15 are used to control the flow of water to the contactors. Preferably, flow would be arranged so that stripping water flows through two parallel banks of two contactors 28, 30 and 32, 34 in series. In this configuration, the isolation valves 154 and 158 are closed; bypass valves 160 and 162 are open and bypass valves 164 and 166 are closed.

Where the total flow of strip water in line 150 is greater than about 1,200 litres per
20 hour the pressure differential across the lumen (water) side of the third, fourth, fifth and sixth contactors 28, 30, 32 and 34 as measured by pressure transmitters 168 and 170 may become unacceptably high. Preferably the pressure as measured at the transmitter 168 on the line 150 should be less than the pressure in the permeate line 50 as measured at the pressure transmitter 70. If this is not possible by fully opening the valves 152 and 156,
25 then the water flow to the contactors may have to be arranged in a parallel configuration. To do this, the valves 152, 154, 156 and 158 are opened; the bypass valves 160 and 162 are closed; and the bypass valves 164 and 166 are opened. In this way the contactor bank containing the third and fourth contactors 28 and 30 passes alcohol rich strip water to a line 172 and the contactor bank containing the fifth and sixth contactors 32 and 34 passes
30 alcohol rich strip water to a line 174. The flow rates in each of the lines 172 and 174 are

- 14 -

monitored by rotameters 176 and 178 respectively and are controlled by the degree of opening of the valves 152, 154, 156 and 158.

It has been found that the efficiency of alcohol extraction in the stripping contactors 28, 30, 32 and 34 is improved if both the permeate and the strip solution have carbon dioxide and oxygen removed therefrom. As described previously, the vacuum pump 40 draws a vacuum on lines 42, 44 and 46 and on the lumen sides of the contactors 24 and 26. Typically the pressure in the vacuum line 42 as measured by a pressure transmitter 180 is about 50 torr.

The system is arranged such that water from the break tank 84 maintains a supply of service (sealing and cooling) water to the vacuum pump 40. The exhaust gases and service water which are ejected by the vacuum pump pass through a line 182 to the surge tank 98 where the gases including the nitrogen strip gas from inlet line 44 and the carbon dioxide and oxygen extracted from the contactors 24 and 26 are expelled to the atmosphere. Some small amounts of alcohol from the permeate in the contactor 26 are also expelled. The water from the line 182 is then available to be used for stripping purposes, so minimising the use of water by the system.

The reference to any prior art in this specification is not, and should not be taken as, an acknowledgment or any form of suggestion that that prior art forms part of the common general knowledge in Australia.

Many modifications will be apparent to those skilled in the art without departing from the spirit and scope of the invention.

DATED this 21st day of December, 2004

WINE NETWORK TECHNOLOGY PTY. LTD.

By its Patent Attorneys

DAVIES COLLISON CAVE

2004907247 21 Dec 2004

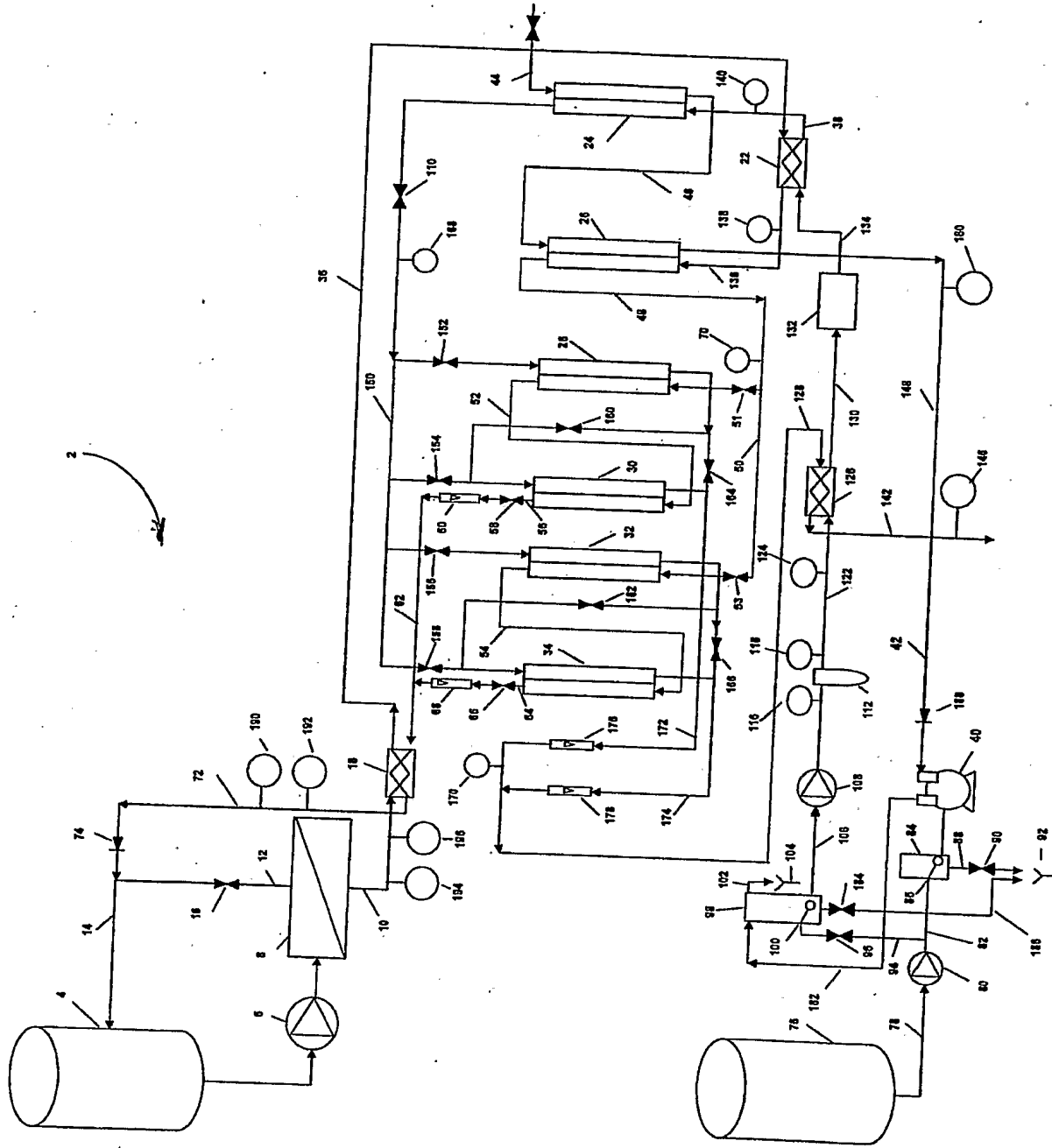


FIG 1